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(54) Title: MULTIFUNCTIONAL TEXTILES

(57) Abstract: The present invention provides a multifunctional textile composition, the textile composition comprises a textile having an antimicrobial functionality; and a chemical agent attached thereto to impart an additional functionality. Suitable additional functionalities include, but are not limited to, waterproof finishing, soil repellent finishing, fire resistance finishing, wrinkle free finishing, anti-UV finishing, and antistatic finishing. By imparting additional functionalities, the textile composition is rendered more versatile.

MULTIFUNCTIONAL TEXTILES

CROSS-REFERENCE TO RELATED APPLICATIONS

This application claims the benefit of U.S. Provisional Patent Application No. 60/280,687, filed March 30, 2001, the teaching of which are incorporated herein by reference in their entirety.

BACKGROUND OF THE INVENTION

Textiles are very important materials related to human life and living that provide decorative and protective functions. Our modern lifestyle has created increased demands on new textile products, ranging from simply comfort feeling to multi-protective functions against various hazardous or severe environments. Today's textile products are not simply apparels, decorations, and classical textiles, but should also be advanced shields to human bodies that are capable of preventing attacks from pathogenic microorganisms, toxic chemicals, flame, UV radiation, and potentially any natural hazards. There are more demands in many specialty textiles in the current textile market, for example, durable and reusable hygienic clothing that can inactivate pathogens and prevent skin infections, antiodor or antimicrobial carpets that can improve indoor air quality, as well medical-use, institutional-use textiles, and the like.

Antimicrobial textiles play an important role in preventing cross transmission of infectious diseases in hospital and healthcare facilities due to the proven evidence that the textiles are major hosting media for the microorganisms (*see*, Sun, G. *et al.*, Durable and Regenerable Antimicrobial Finishing of Fabrics with a New Hydantoin Derivative, *Industrial Engineering Chemistry Research*, Vol. 41, 1016-1021. 2001; Worley, S. D. *et al.*, (1996) "Biocidal Polymers" Trends in *Polymer Science*. V4, p. 364-370; and Rigby, A.J. *et al.*, (1993), *Textile Horizons*, Dec., 42-46).

These antimicrobial materials can be divided into two categories based on their abilities to combat microorganisms, *i.e.*, biocidal and biostatic functions. Biocidal functions refer to the complete inactivation of microorganisms on the materials or total kill, while biostatic properties indicate the inhibition of growth of microorganisms by the materials or partial kill. Based on these definitions, bio-protective clothing should be made of biocidal fabrics instead of the ones in the second category because of the specific functions

required for the protection. Biostatic fabrics would be more appropriate for aesthetic and hygienic type applications of textile products, as well as used in antiodor textiles.

In recent years, there has been the development of innovative technologies that prepare durable functional textiles, including biocidal clothing. For example, cellulose related materials have been generated by using covalent bonding (*see*, Sun, G., *et al.*, *Textile Chemist and Colorist*, **30(6)**:26 (1998); Sun, G., *et al.*, *Textile Chemist and Colorist*, **31(5)**:31 (1999)); Sun, G., *et al.*, *Textile Chemist and Colorist*, **31(1)**:21-24 (1999)). However, due to the lack of reactive groups in most synthetic fibers, there exist limited practical options *i.e.* few intermolecular interactions to achieve durable functions on polymers.

Among the currently investigated biocidal materials, N-halamines have been shown to provide almost instant and total kill of a wide range of microorganisms. (*see*, Worley, S. D. et al., *Trends Polym. Sci.*, 11:364 (1996)). There are many advantages associated with using N-halamine structures. First, they are stable in long-term use and storage over a wide temperature range. Second, they are regenerable when activity is lost due to normal use patterns. (*see*, Sun, G. *et al.*, *Polymer*, 37:3753 (1996); Worley, S. D. *et al.*, *The Polymeric Materials Encyclopedia*, 1, A-B, p. 550 (1996); Sun, G. *et al. Water Res. Bull.*, 1996, 32:793 (1996)). More recently, N-halamine materials have been incorporated into cellulose-containing fabrics. (*see*, Bickert, J. R. *et al.*, *International Conference on Safety & Protective fabric'98*, 1998, p 1; Sun, G. et *al.*, *Textile Chem. Colorist*, 6:26 (1998); Sun, G. *et al.*, *Textile Chem. Colorist*, 31:21 (1999)). Results indicate that as little as 1% (wt) add-on of halamine structures provide powerful biocidal efficacy (6-7 log reduction) against the most common pathogens, at a contact time of two minutes.

U.S. Patent No. 5,882,357, issued to Sun *et al.*, on March 16, 1999, discloses durable and regenerable microbiocidal textiles and methods for preparing the same. The microbiocidal textiles are prepared using a wet finishing process to covalently attach a heterocyclic N-halamine to a cellulose-based material or other polymeric material. The biocidal activity of the textiles can be regenerated by washing with a halogenated solution.

Moreover, PCT Publication WO 00/15897 published March 23, 2000, to Sun *et al.*, discloses durable and refreshable antimicrobial polymers such as textiles, that have excellent colorfastness and washfastness. The textiles are suitable for sportswear, antiodor carpets, films, plastics, toys and medical uses. In that invention, dye molecules are used as connectors or bridges between the textile and antimicrobial agents.

In addition, U.S. Application No. 09/662,999, entitled "Antimicrobial Polymers," to Sun *et al.*, filed September 15, 2000, describes antimicrobial polymers

compositions having a functional monomeric unit; and an antimicrobial agent attached to the functional monomeric unit. The monomeric unit preferably includes a functional group such as a carboxylate group, a sulfonate group, an oxide group, an alkoxide, a phosphate or a phosphonate group. The antimicrobial polymers are preferably antimicrobial textiles, which can be used in a wide variety of applications. Suitable applications include surgeon's gowns, caps, masks, surgical covers, patient drapes, carpeting, bedding material, underwear, socks, sportswear and healthcare uniforms.

U.S. Patent No. 6,020,491, issued to Wonley *et al.*, on February 1, 2000, discloses cyclic amine monomers and polymers that are used to form biocidal N-halamine polymers. The polymers are useful as disinfectants for potable water, swimming pools, hot tubs, industrial water systems, cooling towers, air-conditioning systems, and the like.

Despite the advances made in the art, there exists a need for new antimicrobial textiles having additional functionality. Textiles having multifunctional features are needed for myriad applications. For example, medical-use clothing may require both waterproofing and antimicrobial properties, while some sportswear should provide both anti-UV and odor-free performance. The development of multifunctional textiles based on antimicrobial technologies is needed. The present invention provides these and other needs.

SUMMARY OF THE INVENTION

Textiles having multifunctional features are needed for medical applications, industrial safety clothing and sportswear use. For example, medical-use clothing may require both waterproofing and antimicrobial properties, while some sportswear should provide both anti-UV and odor-free performance. As such, in one embodiment, the present invention provides a multifunctional textile composition, the textile composition comprising: a textile having an antimicrobial functionality; and a chemical agent attached thereto to impart an additional functionality. Suitable additional functionalities include, but are not limited to, waterproof finishing, soil repellent finishing, fire resistance finishing, wrinkle free finishing, anti-UV finishing, and antistatic finishing. By imparting additional functionalities, the textile composition is rendered more versatile.

In another embodiment, the present invention provides a process for preparing the multifunctional textiles of the present invention. The process comprises (a) preparing a textile having an antimicrobial functionality to generate an antimicrobial textile; and (b) treating the antimicrobial textile with a chemical agent to impart an additional functionality, thereby preparing the multifunctional textile.

In still yet another embodiment, the present invention provides a garment or article comprising: an antimicrobial functionality; and a chemical agent attached thereto to impart an additional functionality. The garment can be for example, a surgeon's gown, a cap, a mask, a surgical cover, a patient drape, and the like. The garment can be prepared for example, using the textiles of the present invention.

Numerous benefits are achieved by way of the present invention over conventional techniques. There are many potential application areas for the new textile materials. For example, the new textile material is not only a physical barrier to microorganisms, in addition, it can provide a disinfectant property as well. This regenerable and reusable biocidal material can replace currently used disposable nonwoven fabrics in hospitals, and serve as a safeguard to medical workers and patients. The antimicroorganism properties of the textile materials of the present invention, particularly the antiodor properties, can impart beneficial properties to apparel products such as underwear, socks, and sportswear. Moreover, the antimicrobial fabrics are advantageous to hotels and institutions for such uses as towels, bedding materials, carpets, and wall covers, as a safeguard in preventing cross-contamination of infectious diseases. Depending upon the embodiment, one or more of these benefits may be achieved.

Various additional objects, features and advantages of the present invention can be more fully appreciated with reference to the detailed description and accompanying drawings that follow.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a simplified diagram of a chemical modification process according to an embodiment of the present invention;

Fig. 2 illustrates examples of heterocyclic N-halamines precursors suitable for use in the present invention;

Fig. 3 is a simplified flow diagram that illustrates a chemical modification process according to an embodiment of the present invention;

Fig. 4 is a simplified diagram of a chemical modification process according to an embodiment of the present invention;

Fig. 5 illustrates various disperse dyes suitable for use according to an embodiment of the present invention;

Fig. 6 illustrates various acid dyes suitable for use according to an embodiment of the present invention.

DETAILED DESCRIPTION OF THE INVENTION AND PREFERRED EMBODIMENTS

I. MULTIFUNCTIONAL TEXTILES

The present invention provides durable multifunctional textiles and fabrics. In one embodiment, the present invention provides a multifunctional textile composition, the textile composition comprising: a textile having an antimicrobial functionality; and a chemical agent attached thereto to impart an additional functionality. The terms "antimicrobial," "microbicidal," or "biocidal" as used herein, refer to the ability to kill at least some types of microorganisms, or to inhibit the growth or reproduction of at least some types of microorganisms. The textiles prepared in accordance with the present invention have microbicidal activity (antimicrobial) against a broad spectrum of pathogenic microorganisms. The textiles have microbicidal activity against representative gram-positive (such as *Staphylococcus aureus*) or gram-negative bacteria (such as *Escherichia coli*) or combinations thereof. In certain preferred aspects, the microbicidal activity of such textiles is readily regenerable.

The term "multifunctional textile" as used herein, refers to a microbiocidal textile as previously defined, comprising an additional functionality. Suitable additional functionalities include, but are not limited to, waterproof finishing, soil repellent finishing, fire resistance finishing, wrinkle free finishing, anti-UV finishing, and antistatic finishing. By imparting additional functionalities, the textile composition is rendered more versatile.

A. ANTIMICROBIAL FUNCTIONALITY

1. Heterocyclic N-Halamines

The multifunctional textiles of the present invention comprise, for example, antimicrobial properties. As discussed in detail herein, the antimicrobial functionality can be imparted in a variety of ways. In a preferred embodiment, the multifunctional textile compositions of the present invention have antimicrobial functionality imparted using heterocyclic N-halamine chemistry. For example, U.S. Patent No. 5,882,357, which issued to

Sun *et al.* on March 16, 1999, and incorporated herein by reference, teaches an antimicrobial textile composition comprising: a textile material such as cellulose, cellulosic-polyester and polyester material; and a heterocyclic N-halamine covalently attached to the textile material. The antimicrobial textile material is both durable and regenerable.

"Heterocyclic N-halamine," as used herein, refers to a 4- to 7-membered ring, wherein at least 3 members of the ring are carbon, and from 1 to 3 members of the ring are nitrogen(s) heteroatom, and from 0 to 1 member of the ring is an oxygen atom, wherein from 0 to 2 carbon members comprise a carbonyl group, and wherein at least 1 to 3 nitrogen atoms are substituted with a hydrogen or hydroxyalkyl group, such as $-CH_2OH$, or a alkoxyalkyl group, such as $-CH_2OCH_3$. At least one ring nitrogen has bonded thereto a halogen atom. In addition, the ring members can be further substituted with alkyl groups, such as methyl, ethyl, and the like or hydroxy groups. Heterocyclic N-halamines are generally disclosed in U.S. Patent No. 5,490,983 issued to Worley, *et al.* on Feb. 13, 1996, the teachings of which are incorporated herein by reference for all purposes.

Figure 1 shows schematically 100 an example of a heterocyclic amine 110 being covalently bonded to cellulose 105 to produce a cellulose-potential biocide moiety 120. After activation with a halogen source 130, a cellulose-biocide is generated 150 (*e.g.*, heterocyclic N-halamines). Precursors of the heterocyclic N-halamines 120 suitable for use in the present invention are exemplified in Fig. 2. Examples include, but are not limited to, monomethylol-5,5-dimethylhydantoin (MDMH), 1,3-dimethylol-5,5-dimethylhydantoin (DMDMH); monomethylolated and dimethylolated derivatives of 2,2,5,5-tetramethyl-1,3-imidazolidin-4-one, 6,6-dimethyl-1,3,5-triazine-2,4-dione, 4,4,5,5-tetramethyl-1,3-imidazolidin-2-one, cyanuric acid and 5,5-dimethylhydantoin; and monomethoxylated and dimethoxylated derivatives of monomethylolated and dimethylolated derivatives of 6,6-dimethyl-1,3,5-triazine-2,4-dione, 4,4,5,5-tetramethyl-1,3-imidazolidin-2-one, cyanuric acid, 5,5-dimethylhydantoin, 2,2,5,5-tetramethyl-1,3-imidazolidin-4-one and mixtures thereof.

With reference to Figure 3, the antimicrobial functionality can be imparted using process 300. This diagram is merely an example, which should not limit the scope of the claims herein. One of ordinary skill in the art would recognize many other variations, modifications, and alternatives. As illustrated in Fig. 3, the process comprising (a) immersing a cellulosic textile in an aqueous treating solution, which comprises a catalyst, a wetting agent and a heterocyclic amine 310; removing the excess treating solution from the cellulosic textile 320; (c) drying the cellulosic textile 330; (d) curing the dried cellulosic textile 340; (e) washing the cured cellulosic textile to remove excess reagents 350; (f) drying

the cellulosic textile to remove water 360; and (g) treating the cellulosic textile with a halogenated aqueous solution to produce a heterocyclic N-halamine, thereby preparing a microbiocidal cellulosic textile 370. The foregoing process is claimed and taught in U.S. Patent No. 6,077,319, which issued to Sun *et al.* on June 20, 2000 and is incorporated herein by reference in its entirety for all purposes. In certain aspects, waterproofing, fire resistant agents and the like, can be added in the finishing bath 310 so as to achieve multifunctional textiles of the present invention.

Advantageously, the foregoing process is executed by the utilization of redox reactions. Thus, the potential biocidal groups 110 can be activated by a common laundering process 130, which will enable users to functionalize the materials at any convenient time. In addition, laundering bleaches such a CLOROX® are household chemicals that normally do not possess harmful effects to wearers and handlers who wash and regenerate the functional fabrics.

In certain instances, a byproduct of producing antimicrobial articles using heterocyclic N-halamine chemistry is the release of free-formaldehyde. To reduce the amount of free-formaldehyde release, in certain aspects, the above process optionally further comprises: (a) immersing the article or textile in an aqueous treating solution which comprises a heterocyclic amine and a polyol; and (b) treating the article with a halogenated solution, thereby rendering the article microbiocidal with a reduction in free-formaldehyde release. Suitable polyols include, but are not limited to, diethylene glycol or ethylene glycol. In certain preferred embodiments, the heterocyclic amine is alkylated or partially alkylated. Such processes are described and taught in U.S. Patent No. 6,241,783, issued to Sun on June 5, 2001, and is hereby incorporated by references in its entirety for all purposes.

2. Polymer:Bridge:Biocide Motif

In other embodiments, the antimicrobial properties can be imparted using a dye or colorant as a bridge between the textile and an antimicrobial agent. The chemistry is illustrated in Figure 4. This schematic diagram 400 is merely an example, which should not limit the scope of the claims herein. One of ordinary skill in the art would recognize many other variations, modifications, and alternatives.

Figure 4 shows an antimicrobial polymer composition 440 comprising: a polymer material having a colorant 420 and an antimicrobial agent or biocide 430 attached to the colorant 420. The colorant 420 acts as a bridge and can be a dye or a pigment. In one

embodiment, the polymer 410 is a textile, such as a fabric. In certain preferred aspects, colorants, such as dyes, are used as connectors, bridges or links 420, to firmly attach the microbicidal agents to the polymer. In other aspects, the dyes contain auxochromes, such as sulfonic, hydroxyl and amino groups that can be used to facilitate color shades and solubility requirements. The antimicrobial agents 430 are amphipathic molecules. Preferably, the antimicrobial agents are quaternary ammonium salts. Such antimicrobial textile can be generated using a process comprising: a) dyeing a polymer with a colorant to form a polymer having the colorant attached thereto; and b) attaching a antimicrobial agent to the colorant, thereby making the polymer antimicrobial. The colorant can be a dye or a pigment.

In certain aspects, the biocide 430 is a quaternary ammonium salt. Suitable quaternary ammonium salts include, but are not limited to, dodecyltrimethyl ammonium bromide (DTAB), N-(3-chloro-2-hydroxypropyl)-N,N-dimethyldodecylammonium chloride, 1,3-Bis-(N,N-dimethyldodecylammonium chloride)-2-propanol, dodecyltrimethyl ammonium chloride (DTAC), N-(1-(2,3-dioleoyloxy)propyl)-N,N,N-trimethylammonium chloride (DOTAP), N-(1-(2,3-dioleyloxy)propyl)-N,N,N-trimethylammonium chloride (DOTMA), dimethyldioctadecyl ammonium bromide (DDAB), N,N-dioleyl-N,N-dimethylammonium chloride (DODAC) and 1,2-dioleoyloxy-3-(N,N,N--trimethylamino)propane chloride (DOTAP). Preferably, the quaternary ammonium salts are dodecyltrimethyl ammonium bromide (DTAB), N-(3-chloro-2-hydroxypropyl)-N,N-dimethyldodecylammonium chloride, 1,3-Bis-(N,N-dimethyldodecylammonium chloride)-2-propanol, cetyl pyridinium chloride (CPC), and benzyldimethylhexadedylammonium chloride (BDHAC).

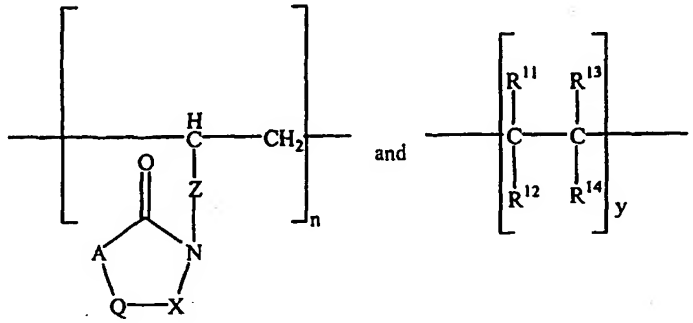
Antimicrobial textiles formed using the polymer:brige:biocide motif are set forth and taught in U.S. Patent Application No. 09/151,891, filed September 11, 1998, published as WO 00/15897 on March 23, 2000, and is hereby incorporated by reference for all purposes.

The refreshing process 480 is the regular laundering practice, which removes cell bodies 450 that are killed by the biocides but are left on the surface. In certain aspects, waterproofing, fire resistant agents and the like, can be added in the finishing bath so as to achieve multifunctional textiles of the present invention.

3. Antimicrobial Heterocyclic Polymers

In certain other embodiments, the antimicrobial functionality can be imparted using heterocyclic vinylic amines, which can be readily polymerized with most acrylic,

substituted-acrylic and vinyl monomers. The polymers thus generated exhibit biocidal efficacy after exposure to a halogen source, such as chlorine bleach. The antimicrobial polymer comprises a mixture of monomeric units having the formulae:



I

wherein:

A is a member selected from the group consisting of NH, N-R^8 and CR^1R^2, wherein R^8 is a halogen;

R^1 and R^2, are each independently selected from the group consisting of optionally substituted (C_1-C_6)alkyl, optionally substituted (C_2-C_6)alkenyl, optionally substituted (C_2-C_6)alkynyl, optionally substituted cycloalkyl, optionally substituted (C_1-C_6)alkoxy, optionally substituted aryl and optionally substituted heteroaryl;

or, R^1 and R^2 and the carbon to which they are bound join to form an optionally substituted carbocyclic or optionally substituted heterocyclic ring;

Q is a member selected from the group consisting of C(O), NH, N-R^9 and CR^3R^4, wherein R^9 is a halogen;

R^3 and R^4, are each independently selected from the group consisting of optionally substituted (C_1-C_6)alkyl, optionally substituted (C_2-C_6)alkenyl, optionally substituted (C_2-C_6)alkynyl, optionally substituted cycloalkyl, optionally substituted (C_1-C_6)alkoxy, optionally substituted aryl and optionally substituted heteroaryl;

or, R^3 and R^4 and the carbon to which they are bound, join to form an optionally substituted carbocyclic or optionally substituted heterocyclic ring.

X is a member selected from the group consisting of C(O)-NR^{10} and CR^6R^7, wherein R^{10} is a member selected from the group consisting of hydrogen, halogen, optionally substituted (C_2-C_6)alkenyl and optionally substituted (C_1-C_6)alkyl;

R^6 and R^7, are each independently selected from the group consisting of optionally substituted (C_1-C_6)alkyl, optionally substituted (C_2-C_6)alkenyl, optionally

substituted (C_2-C_6)alkynyl, optionally substituted cycloalkyl, optionally substituted (C_1-C_6)alkoxy, optionally substituted aryl and optionally substituted heteroaryl;

or, R^6 and R^7 and the carbon to which they are bound join to form an optionally substituted carbocyclic or optionally substituted heterocyclic ring;

Z is a member selected from the group consisting of optionally substituted (C_1-C_3)alkylene, C(O), or a single bond;

R^{11} is a member selected from the group consisting of hydrogen, halogen, hydroxyl, cyano, (C_1-C_6)alkyl, (C_2-C_6)alkenyl, (C_1-C_6)alkoxy, (C_1-C_6)alkylcarbonyl, (C_1-C_6)alkylcarboxyl, aldehydo, amido, aryl and heterocyclyl;

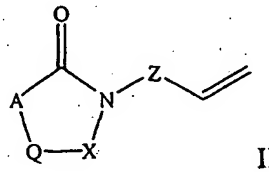
R^{12} is a member selected from the group consisting of hydrogen, halogen, hydroxyl, cyano, (C_1-C_6)alkyl, (C_2-C_6)alkenyl, (C_1-C_6)alkoxy, (C_1-C_6)alkylcarbonyl, (C_1-C_6)alkylcarboxyl, aldehydo, amido, aryl and heterocyclyl;

R^{13} is a member selected from the group consisting of hydrogen, halogen, hydroxyl, cyano, (C_1-C_6)alkyl, (C_2-C_6)alkenyl, (C_1-C_6)alkoxy, (C_1-C_6)alkylcarbonyl, (C_1-C_6)alkylcarboxyl, aldehydo, amido, aryl and heterocyclyl;

R^{14} is a member selected from the group consisting of hydrogen, halogen, hydroxyl, cyano, (C_1-C_6)alkyl, (C_2-C_6)alkenyl, (C_1-C_6)alkoxy, (C_1-C_6)alkylcarbonyl, (C_1-C_6)alkylcarboxyl, aldehydo, amido, aryl and heterocyclyl; and

n and y are each independently an integer from 1 to 250 inclusive. In certain preferred aspects, n is 1 and y is 1. In equally preferred aspects, A is NH and Z is CH_2. The biocidal heterocyclic vinylic amines are taught in U.S. Patent Application No. 09/535,348, filed March 24, 2000, published as WO 01/72715 on October 4, 2001, and is hereby incorporated by reference in its entirety for all purposes.

Vinyl monomers suitable for use in the present invention include, but are not limited to, an acrylic monomer, a monofunctional vinyl monomer, a polyfunctional vinyl monomer and mixtures thereof. The polymerization reaction proceeds with a compound set forth below:



II

wherein A, Q X, N and Z were defined above previously, and at least one other existing vinyl monomer, optionally in the presence of a free radical initiator. The

reaction can take place in bulk, an aqueous solution, a suspension, an organic solvent, or emulsion.

Once formed, the polymers can be made biocidal by reacting the corresponding unhalogenated polymers, with a halogen source. Suitable halogenating agents include, but are not limited to, calcium hypochlorite, sodium hypochlorite (*e.g.*, CLOROX®), N-chlorosuccinimide, N-bromosuccinimide, sodium dichloroisocyanurate, trichloroisocyanuric acid, tertiary butyl hypochlorite, N-chloroacetamide, N-chloramines, N-bromamines, and the like. In certain aspects, waterproofing, fire resistant agents and the like, can be added in the finishing bath so as to achieve multifunctional textiles of the present invention.

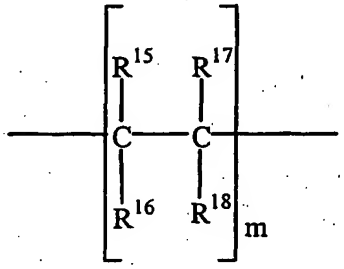
The halogenation of the unhalogenated polymers can be accomplished in aqueous media or in mixtures of water with common inert organic solvents such as methylene chloride, chloroform, and carbon tetrachloride, or in inert organic solvents themselves, at room temperature. Those of skill in the art will know of other solvents or solvent mixtures suitable for use in the present invention. In certain instances, the unhalogenated polymers can be a previously-utilized cyclic N-halamine polymer that needs to be regenerated due to inactivation of the N-halamine moieties. As used herein, "halogenating" or "halogenated" polymers refers to partially as well as fully halogenated. Preferred halogens are chlorine and bromine.

4. Antimicrobial Polymers

In another embodiment, the antimicrobial functionality of the multifunctional textile composition of the present invention can be imparted using other antimicrobial polymers. For example, reactive or functional groups in polymers (*e.g.* acrylics and nylons) such as anionic groups, can be employed as a point of attachment for antimicrobial agents. Dye molecules having a complementary functional group, such as a cationic group, can penetrate into and reside in the polymers to form strong interactions, such as ionic interactions, with their counterparts (*e.g.*, anionic group). The present invention provides a multifunctional textile having an antimicrobial polymer composition comprising: a) a polymer having a functional monomeric unit; and b) an antimicrobial agent attached to the functional monomeric unit. The functional monomeric unit serves as a point of attachment for interaction with the antimicrobial agent. In certain instances, the antimicrobial composition employs ionic interactions between polymers such as acrylic polymers, and

polyamides, and antimicrobial agents such as quaternary ammonium salts. In certain aspects, the finishing conditions, polymer morphology, and structure of the antimicrobial agents play important roles in achieving durable performance of the compositions. Preferably, the composition comprises a synthetic organic polymer such as an acrylic polymer, or polyamides (nylons and Aramid), or cationic dyeable polyester. The polymer can be a fiber woven into a textile.

In a preferred embodiment, the antimicrobial polymer comprises at least one functional monomeric unit having the formula:



III

wherein:

R^{15}, R^{16} and R^{17} are each independently, a member selected from the group consisting of hydrogen, halogen, hydroxyl, cyano, $(C_1\text{-}C_6)$alkyl, $(C_2\text{-}C_6)$alkenyl, $(C_1\text{-}C_6)$alkoxy, $(C_1\text{-}C_6)$alkylcarbonyl, $(C_1\text{-}C_6)$alkylcarboxyl, aldehydo, amido, aryl and heterocyclyl;

R^{18} is a member selected from the group consisting of $-CO_2^- X^{1+}$, $SO_3^- X^{1+}$, $-O^- X^{1+}$, $-PO_4^{-2} Z X^{1+}$ and $-PO_3^{-2} Z X^{1+}$;

X^1 is a member selected from the group consisting of a quaternary ammonium salt, a basic dye, metal ions such as Ag^+, Au^+, Cu^{++} and the like, and a mixtures thereof;

Z^1 is a member selected from the group consisting of hydrogen and an alkaline earth metal; and m is an integer from 1 to 250 inclusive. In certain preferred embodiments, such antimicrobial polymers comprise a long-chain synthetic acrylic polymer or fiber comprising at least 35% by weight of acrylonitrile units or fibers containing other reactive groups such as nylons.

A wide variety of polymers can be used in the present invention. Suitable polymers include, but are not limited to, fibers from plants, polymers from animals, natural organic polymers, synthetic organic polymers and inorganic substances. In a preferred aspect, synthetic organic polymers such as acrylic polymers and polyamides are used. In certain aspects, the acrylic polymers suitable for use in the present invention have a number

average molecular weight of about 40,000 to about 60,000 or about 1000 to about 1500 repeat units. The weight average molecular weight is about 90,000 to about 140,000, with the polydispersity index between about 1.5 to about 3.0.

Moreover, in certain embodiments, the polymer is a plurality of polymers. Suitable plurality of polymers include, but are not limited to, fibers, films, textiles and plastics. In preferred aspects, the antimicrobial fibers are acrylic fibers or polyamides fibers. As used herein, the term "acrylic fiber" means any manmade fiber derived from acrylic resins comprising a minimum of 85% acrylonitrile, contained therein. Acrylic fiber is a manufactured fiber in which the fiber forming substance is any long-chain synthetic polymer comprising at least 85% by weight of acrylonitrile units $(-CH_2-CH[CN]-)_x$. As used herein the term "modacrylic fiber" means a fiber having less than 85% by weight of acrylonitrile units, but at least 35% by weight of acrylonitrile units.

In a preferred embodiment, the acrylic fibers used in the present invention are made from acrylonitrile and at least one other functional monomer. The functional monomers have a functional group, preferably an ionic functional group. Suitable functional groups include, but are not limited to, a carboxylate ($-CO_2^-$) group, a sulfonate (SO_3^-) group, a hydroxide (-OH) group, an alkoxide ($-RCH_2O^-$) group, a phosphate ($-PO_4^{-2}$) group and a phosphonate ($-PO_3^{-2}$) group. In a preferred aspect, the functional monomer comprises a negatively charged functional monomer such as a sulfonate (SO_3^-) group. Functional monomers comprising a sulfonate group include, but are not limited to, sodium styrenesulfonate, sodium methyallyl sulfonate and sodium sulfophenyl methallyl ether.

Suitable acrylic fibers are produced by various manufactures. Suitable acrylic fibers for use in the present invention include, but are not limited to, MicroSupreme®, Cresloft™, Creslan® Plus, BioFresh™, WeatherBloc™ (commercially available from Sterling Fibers, Inc.); Dralon™ (commercially available from Bayer Inc.) Acrilan®, Bounce-Back®, Duraspun®, Pil-Trol®, Sayelle®, Sno-Brite™, The Smart Yarns®, Wear-Dated® and Wintuk® (commercially available from Solutia Inc.). Other acrylic fibers include Orlon®, Acrilin® acrylic, Dolan®, Dralon®, Vinyon N®, Dynel®, Verel®, SEF modacrylic®. Polyamide fibers include, but are not limited to, all nylon fibers such as nylon 6, nylon 66, Nomex, Kermel, and Kevlar. Those of skill in the art will know of other manufactures and trade names of acrylic fibers suitable for use in the present invention.

In certain aspects, waterproofing, fire resistant agents and the like, can be added in the finishing bath so as to achieve multifunctional textiles of the present invention.

B. ADDITIONAL FUNCTIONALITIES

In addition to antimicrobial features, the present invention provides textiles having additional functionalities. Suitable additional functionalities include, but are not limited to, waterproof finishing, soil repellent finishing, fire resistance finishing, wrinkle free finishing, anti-UV finishing, and antistatic finishing. By imparting additional functionalities, the textile composition is rendered more versatile.

1. Durable Waterproofing and Soil Repellent Finishing

In certain aspects, the textiles of the present invention have functionalities of waterproofing and soil repellency. Waterproofing can be imparted by treating the fabrics in a finishing bath containing a chemical agent such as fluorocarbons, silicones, or other waterproofing agents. Preferably, hydrophilic fabrics or materials are used. These hydrophilic fabrics include, but are not limited to, cotton, cotton containing fabrics, wool, wool containing fabrics, silk and silk containing fabrics.

Soil-repellent finishing can be used to prevent the fabrics and materials from soiling easily. Preferred soil-repellant chemical agents are fluorocarbon chemicals. Suitable waterproofing and soil-repellent chemical agents include, but are not limited to, 3M Protective Chemical® (commercially available from 3M), SEQUAPEL® (commercially available from Omnova Solutions Inc.), FREE PEL® (commercially available from BF Goodrich Performance Materials), BARPEL® (commercially available from Apollo Chemical Corp.), GLO-PEL® and GLO-GUARD® (commercially available from Glo-Tex International Inc.).

2. Fire-resistant finishing

In certain aspects, the textiles of the present invention have a fire resistant functionally. Fire-resistant treatments are used advantageously for home furnishing and some apparel textiles. The function can be achieved by treating the materials with flame retardant chemicals such as phosphorus, nitrogen, bromine, and antimony containing compounds, and combinations thereof. Examples include, but not limited to, GUARDEX PFR® (commercially available from Glo-Tex International Inc.), BARFIRE® (commercially available from Apollo Chemical Corp.), PYROVATEX CP® (commercially available from Ciba Specialty Chemicals) and PYROSAN® (commercially available from BF Goodrich Performance Materials).

3. Wrinkle free finishing

In another aspect, the textiles of the present invention have wrinkle free functionality. A wrinkle free functionality is an important feature for pure cotton and cotton blend fabrics, and fabrics containing other natural fibers such as wool and silk. Examples include, but are not limited to, FREEREZ® (commercially available from BF Goodrich Performance Materials) and PERMAFRESH® (commercially available from Omnova Solutions Inc.).

4. Anti-UV finishing

In certain other aspect, the textiles of the present invention have anti-UV functionality. Anti-UV functions are advantageously used for protection of both wearers and textile materials. Both employ similar chemicals, *i.e.* ultraviolet absorbents or light stabilizers. Typical examples include, but are not limited to, CBAFAST® (commercially available from Ciba Specialty Chemicals), SUNLIFE® (commercially available from NICCA USA Inc.), and ORCO SUNGUARD® (commercially available from Organic Dyestuffs Corp).

5. Antistatic finishing

In certain other aspects, the textiles of the present invention have antistatic functionality. Antistatic treatment advantageously removes static charges built up on surfaces of textiles. The mechanism is to create a conductive layer on the surface, so charges cannot accumulated upon friction between surfaces of textile materials. Antistatic function is required for some institutional clothing and textiles, such as garments worn by petroleum workers, firefighters, and micro-electronic workers. Examples of antistatic agents include, but are not limited to, BARSTAT® (commercially available from Apollo Chemical Corp.), ZEROSTAT® (commercially available from Ciba Specialty Chemicals), and DOW CORNING FLUID® (commercially available from Dow Corning Corp).

II. TEXTILES

The textiles suitable for the present invention include, but are not limited to, naturally occurring fibers from plants, such as cellulose, cotton, linen, hemp, jute and ramie. They include polymers from animals, based upon proteins and include, but are not limited to,

wool, mohair, vicuna and silk. Textiles also include manufactured fibers based upon natural organic polymers such as, rayon, lyocell, acetate, triacetate and azlon. Textiles suitable for use in the present invention include synthetic organic polymers which include, but are not limited to, acrylic, aramid, nylon, olefin, polyester, spandex, vinyon, vinyl and graphite. Textiles also include inorganic substances such as glass, metallic and ceramic.

Considering both antibacterial and mechanical properties of the finished textiles prepared using the methods and compositions set forth herein, those of skill will readily appreciate that such finished textiles can advantageously be used in the preparation of the following articles/garments: surgeon's gowns, caps, masks, surgical cover, patient drapes, carpeting, bedding materials, underwear, socks, uniforms, and the like. Those of skill in the art will readily appreciate that the finished textiles of the present invention can also advantageously be used for a variety of other purposes, such as in hotel-use towels, bedding materials, hygienic products, in various clothing to protect against pesticides and other toxic chemicals, and the like.

Numerous applications for the multifunctional textiles of the present invention exist. For instance, the multifunctional textiles can provide biocidal protective clothing to personnel in the medical area as well as in the related healthcare and hygiene area. The regenerable and reusable biocidal materials can replace currently used disposable, nonwoven fabrics as medical textiles, thereby significantly reducing hospital maintenance costs and disposal fees. The multifunctional textiles of the present invention can be advantageously used for women's wear, underwear, socks, sportswear and other hygienic purposes. In addition, the multifunctional properties can be imparted to carpeting materials to create odor-free and germ-free carpets. Moreover, all germ-free environments, such as required in biotechnology and pharmaceutical industry, would benefit from the use of the microbicidal textiles of the present invention to prevent any contamination from air, liquid, and solid media.

The multifunctional textiles are effective against all microorganisms. Such microorganisms include, for example, bacteria, protozoa, fungi, viruses and algae. Moreover, the multifunctional textiles described herein can be employed in a variety of disinfecting applications, such as water purification. They will be of importance in controlling microbiological contamination or growth of undesirable organisms in the medical and food industries.

III. METHODS

One or more beneficial functionalities can be imparted to the textiles of the present invention. For example, the durable and regenerable antimicrobial functions can be incorporated to textile materials possessing UV-protection with one or more of the above regular wet finishing processes.

In certain aspects, fabrics can be treated in one finishing bath containing the biocidal agent (such as DMDMH) and other functional agents (such as anti-UV chemicals or flame retardants). In other aspects, fabrics are first treated with the antimicrobial agents and then treated with other functional agents. In still further aspects, fabrics are treated with other functional agents and then with the antimicrobial agents. All such treatment sequences and combinations are contemplated in the present invention.

In a preferred method, the chemical finishing of fabrics by biocidal or potential biocidal agents and other functional compounds are carried out concurrently in a wet finishing process. The effects of concentrations of finishing agents, catalysts, carrier of biocides, and other chemicals are optimized in terms of the best biocidal and mechanical properties as well as the best economical concerns.

In a preferred method, an aqueous treating solution comprises a heterocyclic amine or a polymer comprising a heterocyclic amine as described above, a wetting agent and a catalyst. In certain aspects, waterproofing, fire resistant agents and the like, can be added in the finishing bath so as to achieve multifunctional textiles of the present invention. As used herein, "wetting agent" refers to a substance that increases the rate at which a liquid spreads across a surface, *i.e.*, it renders a surface nonrepellent to a liquid. Examples of suitable wetting agents include, but are not limited to, Triton X-100® (Sigma Chemical Co., St. Louis, MO), SEQUAWET® (Sequal Chemical Inc., Chester, SC), and AMWET® (American Emulsions Co., Dalton, GA). Other wetting agents suitable for use in the present invention will be known to and used by those of skill in the art. As used herein, "catalyst" refers to a substance which augments the rate of a chemical reaction without itself being consumed. Suitable catalysts for use in the present invention include, but are not limited to, the following: magnesium salts, zinc salts and ammonium salts. In a presently preferred embodiments, the catalyst employed is one of the following: $MgCl_2$, $Mg(NO_3)_2$, $Zn(NO_3)_2$ and NH_4NO_3.

Those of skill in the art will readily appreciate that the concentration of the various components of the aqueous treating solution can be widely varied depending upon the

particular components employed and the results desired. Typically, the heterocyclic amine is present at a concentration of at least about 0.2%. More typically, the heterocyclic amine is present at a concentration ranging from about 0.2% to about 20%, more preferably at a concentration ranging from about 0.5% to about 10% and, more preferably at a concentration ranging from about 1% to about 5%. It will be readily apparent to those of skill in the art that higher heterocyclic amine concentrations (*e.g.*, 50%) can be employed, but such higher concentrations are not required to impart microbiocidal activity. Again, suitable microbiocidal activity can be imparted using a heterocyclic amine concentration as low as about 0.2%. The wetting agent is typically present at a concentration ranging from about 0.1% to about 3% and, more preferably, at a concentration ranging from about 0.2% to about 1%. The concentration of the catalyst employed will depend on the concentration of the heterocyclic amine employed. Typically, the ratio of heterocyclic amine to catalyst present will range from about 10:1 to about 5:1. The pH of the aqueous treating solution will typically range from a pH of about 2 to about 6 and, more preferably, from a pH of about 2.5 to about 4.5.

Those of skill in the art will readily appreciate that other additives can be incorporated into the aqueous treating solution to impart favorable characteristics to the cellulosic, cellulosic/polyester or polyester textile. Such additives can include softeners and waterproofing agents which are known to and used by those of skill in the art.

In carrying out step 310 in Figure 3, the textile used may be roving, yarn or fabric regardless of whether spun, knit, or woven, or may be nonwoven sheets or webs. Moreover, the textile may be made of cellulosic fibers, polyester fibers or blends of these. In addition, other polymer materials having reactive functional groups (*e.g.*, —OH groups) can be used. Such polymer materials include, but are not limited to, polyvinyl alcohol (PVA), starches and proteins. In wetting the textile in the finishing or treating bath, ordinary textile equipment and methods suitable for batchwise or continuous passage of roving, yarns or fabrics through an aqueous solution may be used, at any speed permitting thorough and uniform wetting of the textile material.

In step 320, the excess aqueous treating solution is removed by ordinary mechanical methods such as by passing the textile between squeeze rolls, by centrifugation, by draining or by padding. In a preferred embodiment, the excess aqueous treating solution is removed by padding.

In step 330, the cellulosic, cellulosic/polyester or polyester textile is dried at a temperature ranging from about 50°C to about 90°C and, more preferably, at a temperature

ranging from about 75°C to about 85°C for a period of time ranging from about 3 to about 8 minutes and, more preferably, for about 5 minutes.

In step 340, the dried cellulosic, cellulosic/polyester or polyester textile is cured at a temperature ranging from about 100°C to about 200°C and, more preferably, at a temperature ranging from about 140°C to about 160°C for a period of time ranging from about 3 to about 8 minutes and, more preferably, for about 5 minutes. The heating can be carried out in an oven, preferably one having a forced draft of air directed at the surface of the textile and exhausting through a vent to remove fumes.

In step 350, the dried cellulosic, cellulosic/polyester or polyester textile is washed. Washing of the treated textile, step (d), may be done with either hot or cold water. The covalent bonds formed are stable, insoluble, and durable to the mechanical agitation, spraying and rubbing that occurs in washing machines or in large scale continuous or batchwise textile washing equipment.

Final drying, step 360, can be carried out by any ordinary means such as oven drying, line drying or tumble drying in a mechanical clothes dryer. A drying temperature of about 80° to about 120°C for about 1 to about 5 minutes is particularly preferred.

Antimicrobial, waterproofing, or fire-resistant functionalities, together with mechanical properties of the fabrics are evaluated by following related AATCC or ASTM test methods. After every five times of washing, the textile materials are regenerated with diluted bleach solutions and tested against microorganisms according to American Association of Textile Chemists and Colorists (AATCC) test methods. The breaking strengths of the regenerated fabrics are examined. A standard commercial dryer is used to evaluate the stability of modified cellulose under different drying conditions after bleaching. Drying temperature is varied from room temperature to high temperature tumble dry. After each drying circle, biocidal properties and tensile strengths of the dried textile materials will be tested again.

As described earlier, in the polymer:brige:biocide motif, colorants, such as dye molecules, are used as connectors or bridges between the textile and the antimicrobial agent. The dye molecules suitable for different synthetic polymers have excellent washfastness and durability. The dyes which can be used include, but are not limited to, an acid dye, a disperse dye, a direct dye and a reactive dye. With reference to Figure 5, various disperse dyes are suitable for use in the present invention. These disperse dyes include, but are not limited to, Disperse Blue 1, Disperse Yellow 7 and Disperse Yellow 9. Those skilled in the art will be aware of various other disperse dyes suitable for use in the present invention.

In a preferred embodiment, an acid dye is used. Suitable acid dyes include, but are not limited to, Acid Black dye, an Acid Blue dye, an Acid Orange dye, an Acid Red dye, an Acid Violet dye, and an Acid Yellow dye. Figure 6 lists various acid dyes suitable for use in the present invention.

In one embodiment, acid dyes are preferably used with Nylon 66, Nylon 6, wool, and silk. In another embodiment, disperse dyes are used with Nylon 66, Nylon 6, Nomex, acetate, triacetate, acrylics, polyester, polypropylene, and blended fabrics. Disperse dyes are also suitable for use in plastic products, such as colored films, toys, computer keyboards and other polymeric products wherein a antimicrobial material is needed.

In certain embodiments, the colorants, such as dye molecules, also contain auxochromes, including, but not limited to, sulfonic groups, hydroxyl groups, quaternary groups and amino functional groups. These auxochromes facilitate the color and solubility requirements of the colorant. In addition, these reactive groups serve to anchor the microbicidal agent. The auxochromes of the dyes serve to chemically modify the polymers such as fibers. These dye molecules then serve as bridges by bringing functional groups onto the polymers, such as fibers, and then linking the biocidal agents. For example, a sulfonate group can form an ionic bond with a cationic species like a quaternary salt, or vice versa. In another example, an amino group and a hydroxyl group are reactive with alkyl halides, epoxide, and acetyl groups. Hence, the dye molecules act as bridges to bring functional groups onto polymers and thereby serving as a point of attachment for the biocidal agents.

In one embodiment, the linkage between the colorant and the antimicrobial agent is an ionic bond formation between a sulfonate anion on an acid dye and an amphipathic molecule, such as a quaternary ammonium salt of a antimicrobial agent. In another embodiment, the linkage is a covalent bond between an amino or hydroxyl group on a disperse dye and an epoxy or alkyl halide structures of a antimicrobial agent. In both cases, quaternary ammonium salts are employed as the antimicrobial agent.

Anionic dyes can interact with positive quaternary ammonium salts due to coulumbic forces, such an interaction can be reflected from the add-on rates of the salts on dyed and undyed fabrics, as well as the weight loss of the sample after repeated washing. Moreover, polyamide structures can form hydrogen bonds or van der Waals interactions with the quaternary ammonium salts, which can result in an add-on of the salts on undyed fabrics. Disperse dyes can covalently link to reactive quaternary ammonium salts, including, but not limited to, N-(3-chloro-2-hydroxypropyl)- N,N-dimethyldodecylammonium chloride.

In this process various colorants can be used. These include, but are not limited to pigments and dye molecules. The colorants are used as connectors between the textile and the antimicrobial agents. The dye molecules suitable for different synthetic polymers have excellent washfastness and durability. The dyes which can be used include, but are not limited to, an acid dye, a disperse dye, a direct dye and a reactive dye. In a preferred embodiment, an acid dye is used. Suitable acid dyes include, but are not limited to, Acid Black dye, an Acid Blue dye, an Acid Orange dye, an Acid Red dye, an Acid Violet dye, and an Acid Yellow dye.

In certain embodiments, the colorants, such as dye molecules also contain auxochromes, such as sulfonic, hydroxyl and amino functional groups. These auxochromes facilitate the color and solubility requirements of the colorant.

Those of skill in the art will readily appreciate that the concentration of the various components of the aqueous treating solution can be widely varied depending upon the particular components employed and the results desired. Typically, the colorant is present at a concentration for an "on weight fabric" (o.w.f.) of about 0.1% to about 15%. More typically, the colorant is present at a concentration ranging from about 0.2% to about 5%, more preferably at a concentration ranging from about 0.5% to about 2%.

The polymer, such as a textile material, is dyed in the colorant solution at about 80°C to about 100°C for about 1 hour to about 3 hours. More typically, about 90°C to about 95°C for about 80 min. to about 90 min. The liquor ratio, which is the ratio of fabric to dye solution (w/w), ranges from about 1:100 to about 1:25, and more preferably about 1:75 to about 1:45, most preferably about 1:50. The pH of the dye bath is adjusted to about pH=1 to about pH=6, more preferably, pH=2.0 to about pH=5 with a weak acid, including, but not limited to, acetic acid. The dyed polymers, such as fabrics, are then washed with an AATCC standard detergent after dyeing and cured. The curing temperature is set to about 90°C to about 150°C, more preferably 100°C to about at 125°C, most preferably 115°C to about 125°C. The cure time is about 5 min to about 30 mins, more preferably 10 min to about 20 minutes.

The antibacterial finishing bath is then prepared by dissolving an antimicrobial agent, such as a quaternary ammonium salt, in distilled water. In certain aspects, waterproofing, fire resistant agents and the like, can be added in the finishing bath so as to achieve multifunctional textiles of the present invention. The pH value is adjusted to about pH=1 to about pH=6, more preferably, pH=2.0 to about pH=5 with a weak acid, such as

acetic acid. The dyed textile is dipped in the antibacterial agent solution, padded to a wet pick up of about 50% to about 120%, more preferably about 60% to about 100% and then cured at an elevated temperature for an extended period. The antimicrobial agent is typically present at a concentration ranging from about 0.1% to about 30% and, more preferably, at a concentration ranging from about 0.2% to about 10%.

In another embodiment, the textiles, such as fabrics, are dyed by an acid dye and then treated in a quaternary ammonium salt solution wherein the treatment is performed in a pressurized dyer or by padding and then curing at a high temperature. Another embodiment involves mixing the acid dye with a quaternary ammonium salt in a bath and directly dyeing or treating the fabric simultaneously.

When using the disperse dye, the treatment can be done by dyeing fabrics first with a disperse dye, then dipping the dyed fabrics in a quaternary ammonium salt solution and padding the fabrics to a wet pick-up rate of 60-120%. The fabrics are cured at about 150-170°C for 5-15 minutes and then washed. Disperse dyes or pigments can be mixed with reactive quaternary ammonium salts under basic conditions (pH >10) in an aqueous solution. The mixture is stirred and warmed for about 30 minutes, and then diluted to 1% o.w.f. Fabrics can be either dipped into the solution, padded at a wet pick-up rate of 60-120%, and cure at 150-170°C for about 5-30 minutes, or immersed in the solution in a pressure dyer, and treated at 120°C, 20 atm pressure for 30 minutes. The fabrics are then washed and dried and ready for testing.

The invention will be described in greater detail by way of specific examples. The following examples are offered for illustrative purposes, and are not intended to limit the invention in any manner.

IV. EXAMPLES

EXAMPLE I

This example illustrates the finishing of fabrics with antibiocidal and waterproofing functionalities.

A finishing bath containing 24 grams of monomethylol-5,5-dimethyl-hydantoin, 4.8 grams magnesium chloride, and 0.6 gram of Triton X-100 (a wetting agent) in 600 milliliters of deionized water was prepared. Waterproofing agents can be added in the finishing baths so as to achieve multifunctional properties on the products. The pH of the finishing bath was adjusted to 3.4 with one milliliter of 0.1 N HCl solution. Then, 140.9

grams of pure cotton fabric (#400 Testfabrics, Inc., Middlesex, NJ) and 141.4 grams of cotton/polyester (35/65) blend fabric (#7409, Testfabrics, Inc., Middlesex, NJ) were dipped in the bath for more than five minutes and padded through a padder with a more than 80% pick-up rate. The fabrics were dipped and padded again, and dried at 80° C for 5 minutes. The fabrics were then cured at 160°C for 5 minutes. Finally, the finished fabrics were machine washed with 90 grams of American Association of Textile Chemists and Colorists (AATCC) Standard Reference Detergent 124 at a low water level and a temperature of about 60°C for 30 minutes. The fabrics were dried and weighed, yielding 42.8 grams (1.35% add-on) of the cotton fabric and 142.4 grams (0.71% add-on) of the cotton/polyester blend fabric. The cotton product exhibited prominent infrared adsorption bands in a KBr pellet at 1718 and 1770 cm^{-1}.

Thereafter, the finished fabrics were washed with a diluted Clorox solution containing about 0.01% active chlorine. Antibacterial properties of the fabrics were tested against representative gram-positive (such as *Staphylococcu aureus* (ATCC 5368)) and gram-negative bacteria (such as *Escherichia coli* (ATCC 2666)).

Example II

This example illustrates the finishing of fabrics with 1,3-dimethylol-5,5-dimethylhydantoin (anitmicrobial) and fire resistance.

A finishing bath containing 48 grams of 1,3-dimethylol-5, 5-dimethylhydantoin (DMDMH), 9.6 grams magnesium chloride and 0.8 gram of Triton X-100 (a wetting agent) in 800 milliliters of deionized water was prepared. Fire resistant agents can be added in the finishing baths so as to achieve multifunctional properties on the products. The pH of the finishing bath was adjusted to 3.1 with 20 milliliters of 0.01 N HCl solution. Then, 144.7 grams of pure cotton fabric (#400 Testfabrics, Inc., Middlesex, NJ) and 143.2 grams of cotton/polyester (35/65) blend fabric (#7409, Testfabrics, Inc., Middlesex, NJ) were dipped in the bath for more than five minutes and padded through a padder with more than an 80% pick up rate. The fabrics were dipped and padded again, and dried at 80°C for 5 minutes. The fabrics were then cured at 160°C for 5 minutes. Finally, the finished fabrics were machine washed with 90 grams of AATCC Standard Reference Detergent 124 at a low water level low and a temperature of about 60°C for 30 minutes. The fabrics were dried and weighed, yielding 147.9 grams (2.22% add-on) of the cotton fabric and 145.5 grams (1.62% add-on) of the cotton/polyester blend fabric. The cotton product exhibited prominent infrared adsorption bands in a KBr pellet at 1718 and 1770 cm^{-1}.

Thereafter, the finished fabrics were washed with a diluted Clorox solution containing about 0.01% active chlorine. Antibacterial properties of the fabrics were tested against representative gram-positive (such as *Staphylococcu aureus* (ATCC 5368)) and gram-negative bacteria (such as *Escherichia coli* (ATCC 2666)).

Example III

This example illustrates the effectiveness of the technology in the biological warfare agent.

The antibacterial textiles produced by using this technology can effectively inactivate *Bacillus subtilis* (an anthrax surrogate). Advantageously, by a contact time of 24 to 48 hours, the fabrics can result in a 97-100% kill to this microorganism. The results are as follows:

Treated non-woven textiles		
1.	50/50 polyester/rayon	100% kill in 48 hours
2.	Treated 100% rayon	97% kill in 24 hours
3.	Treated 30/70 rayon/polypropylene	99.9% kill in 24 hours
Treated woven textiles		
1.	100% cotton canvas	99.9% kill in 48 hours

Example IV

This example illustrates the effectiveness of antibacterial and water repellent treatment.

Bleached and desized cotton print cloth #400 and cotton/polyester (35/65) #7409, supplied by Testfabrics Inc., were used. Water and oil repellent Sequapel Wor were provided by Sequa Chemicals, Inc. The fabrics were finished by using general wet processes, and pad-dry-cure. A finishing bath containing 6% of 1,3-dimethylol-5, 5-dimethylhydantoin (DMDMH), 2% Sequapel Wor agent, 1.5% of magnesium chloride and 0.02% of Triton X-100 (a wetting agent) in 800 milliliters of deionized water was prepared. The pH of the finishing bath was adjusted to 3.5-4 by adding citric acid. Then, pure cotton fabric (#400 Testfabrics, Inc., Middlesex, NJ) and cotton/polyester (35/65) blend fabric (#7409, Testfabrics, Inc., Middlesex, NJ) were dipped in the bath for more than five minutes and padded through a padder with more than an 80% pick up rate. The fabrics were dipped and

padded again, and dried at 80°C for 5 minutes. The fabrics were then cured at 160°C for 5 minutes. Finally, the finished fabrics were machine washed with 90 grams of AATCC Standard Reference Detergent 124 at a low water level and a temperature of about 60°C for 30 minutes. The fabrics were dried and weighed, with 3.52% add-on on the pure cotton and 2.17% add-on on the polyester/cotton blend.

Table 2. Water Droplet Angles on Treated Fabrics

Treatment	Washes	Fabric[1]	1 min	10min	20min	30min	40min	60min	90min
2% WR + 6%DMDMH	0	400	65	70	73	80	90	99	125
	0	7409	65	70	73	77	84	90	120
2% WR + 6%DMDMH	5	400	63	79	87	87	98	112	140
	5	7409	63	68	71	75	81	89	108
2% WR + 6%DMDMH	20	400	150	180	180	180	180	180	180
	20	7409	92	92	103	106	111	131	175
2% WR + 6%DMDMH	50	400	165	180	180	180	180	180	180
	50	7409	108	140	151	162	180	180	180

[1]400 Pure Cotton; 7409 Cotton/Polyester Blend

Table 3.Antibacterial Results of Treated Fabrics Through 50 washes

Durability Treatment	Fabric	Biocidal results of fabrics treated with 2% WR & 6% DMDMH	
		E. Coli	*S. Au*
W0 bleach	400	6 log	6 log
	7409	6 log	6 log
W5 bleach	400	6 log	6 log
	7409	6 log	6 log
W10 bleach	400	6 log	6 log
	7409	6 log	6 log
W15 bleach	400	6 log	6 log
	7409	6 log	6 log
W20 bleach	400	6 log	6 log
	7409	6 log	6 log
W25 bleach	400	6 log	6 log
	7409	6 log	6 log
W30 bleach	400	6 log	6 log
	7409	6 log	6 log
W35 bleach	400	6 log	6 log
	7409	6 log	6 log
W40 bleach	400	6 log	6 log
	7409	6 log	6 log
W45 bleach	400	6 log	6 log
	7409	6 log	6 log
W50 bleach	400	6 log	6 log
	7409	6 log	6 log

Advantageously, after 50 machine washings, the treated fabrics were still maximally biocidally efficacious.

Example V

This example illustrates the preparation of antibacterial high performance fabrics Nomex, Kevlar, and Kermel (aromatic imide-amide) fabrics treated by using the following method.

The finishing bath includes 3% 3-allyl-5,5-dimethylhydantoin (ADMH), 2% poly(ethylene glycol)-diacrylate (PEG-DIA), 1.5% of a commercial softener, and 0.5% of the initiator (such as benzoyl peroxide (BPO) or 2,2'-Azobisisobutyronitrile(AIBN)). The fabrics were dipped-padded twice at a 100% wet pick-up, dried at 50 °C for 5 min, cured at 140 °C for 5 min, washed, dried at 60 °C for 24 h, and stored in a condition room (25 °C, 65% RH) for 48 h to reach constant weight. Percentage graft was calculated from the relation:

$$\text{Graft \%} = (W_2 - W_1) / W_1 \times 100 \qquad (1)$$

wherein W_1 and W_2 are the weights of the original and the grafted fabrics, respectively.

Halogenation

To transform the hydantoin structure in the grafted samples to N-halamines, the grafted fabric was immersed in a diluted bleach containing 3000 ppm active chlorine (bath ratio is 50:1) at room temperature for 30 min, washed thoroughly with a large excess of distilled water, and air dried. The active chlorine content of the fabric was determined by a modified titration method. About 0.3 gram of the treated fabric was cut into small pieces, treated with 30 mL of 0.001 N sodium thiosulfate solution containing 0.05 wt% of a non-ionic wetting agent (Triton X-100) at room temperature under constant stirring over night. The excess sodium thiosulfate was titrated with a 0.001 N iodine solution. Un-chlorinated grafted fabrics were also titrated by using the same methods as controls. Available active chlorine of the bleached grafted fabric was then calculated from equation 2.

$$M_{Cl} = 10^{-6} \times (V_2 - V_1) / W \qquad (2)$$

Where V_1 and V_2 represent the volumes (mL) of iodine solution used in the titration of the sodium thiosulfate solutions treating the samples and the controls, respectively; and W was the weight (g) of the bleached grafted fabric.

Table 4. Percentage Reduction of *E. coli* and *S. aureus* After Different Contact Time (Bacteria concentration: 10^6~ 10^7 CFU/mL)

Fabric	Graft%	Mcl X 10^5 (mol/g)	*E. coli*				*S. aureus*			
			10 min	30 min	60 min	120 min	10 min	30 min	60 min	120 min
Nomex	4.6	1.22	UD*	99.9	99.9999	99.9999	99	99.9999	99.9999	99.9999
Kemel	2.3	0.34	UD	90	99.9	99.9	UD	99.9	99.99	99.999
PBI/Kevlar	2.8	0.41	UD	UD	99.9	99.9	UD	99.9999	99.9999	99.9999

*: no reduction was detected.

Table 5. Percentage Reduction of the bacteria after Washing at a Contact Time of 60 min (Bacteria concentration: 10^6~ 10^7 CFU/mL). All the samples were tested with machine washing following AATCC Test Method 124. AATCC standard reference detergent 124 was used in all the machine-washing tests).

Wash times	Nomex			Kermel			PBI/Kevlar		
	M_{Cl} x10^5 (mol/g)	E. *coli*	S. *aureus*	M_{Cl} x10^5 (mol/g)	E. *coli*	S. *aureus*	M_{Cl} x10^5 (mol/g)	E. *coli*	S. *aureus*
0	1.22	99.9999	99.9999	0.33	99.9	99.999	0.41	99.9	99.9999
5	1.20	99.9999	99.9999	0.28	99.9	99.99	0.41	99.9	99.99
15	0.63	99.9999	99.999	0.23	99.9	99	0.37	99.9	99
30	0.27	99.9	99.99	UD*	90	90	0.20	90	99
50	UD*	90	90	UD*	UD*	UD*	UD*	UD*	UD*
50**	1.14	99.9999	99.9999	0.29	99.9	99.999	0.43	99.9	99.9999

*: no reduction was detected, and **: these samples were re-bleached after 50 times of washing.

It is to be understood that the above description is intended to be illustrative and not restrictive. Many embodiments will be apparent to those of skill in the art upon reading the above description. The scope of the invention should, therefore, be determined not with reference to the above description, but should instead be determined with reference to the appended claims, along with the full scope of equivalents to which such claims are entitled. The disclosures of all articles and references, including patent applications and publications, are incorporated herein by reference for all purposes.

WHAT IS CLAIMED IS:

1. A multifunctional textile composition, said textile composition comprising:

a textile having an antimicrobial functionality; and

a chemical agent attached thereto to impart an additional functionality.

2. The multifunctional textile composition of claim 1, wherein said additional functionality is a member selected from the group consisting of a waterproof finishing, soil repellent finishing, fire resistance finishing, wrinkle free finishing, anti-UV finishing, and antistatic finishing.

3. The multifunctional textile composition of claim 2, wherein said additional functionality is a waterproof finishing.

4. The multifunctional textile composition of claim 3, wherein said waterproofing finishing is imparted with an agent selected from the group consisting of a fluorocarbon agent and a silicone agent.

5. The multifunctional textile composition of claim 2, wherein said additional functionality is a fire resistant finishing.

6. The multifunctional textile composition of claim 5, wherein said fire resistance finishing is imparted with a agent selected from the group consisting of a phosphorus agent, a nitrogen agent, a bromine agent, an antimony agent and combinations thereof.

7. The multifunctional textile composition of claim 2, wherein said additional functionality is a wrinkle free finishing.

8. The multifunctional textile composition of claim 2, wherein said additional functionality is an anti-UV finishing.

9. The multifunctional textile composition of claim 8, wherein said anti-UV finishing is imparted with agent selected from the group consisting of an ultraviolet absorbent or a light stabilizer.

10. The multifunctional textile composition of claim 2, wherein said additional functionality is an antistatic and softening finishing.

11. The multifunctional textile composition of claim 1, wherein said textile is a member selected from the group consisting of cellulosic, cellulosic-polyester, polyester, nylon, polypropylene, acrylics, cotton, wool, silk, polyamide, aramid, olefin, spandex, vinyon, vinyl, graphite, and combinations and blends thereof.

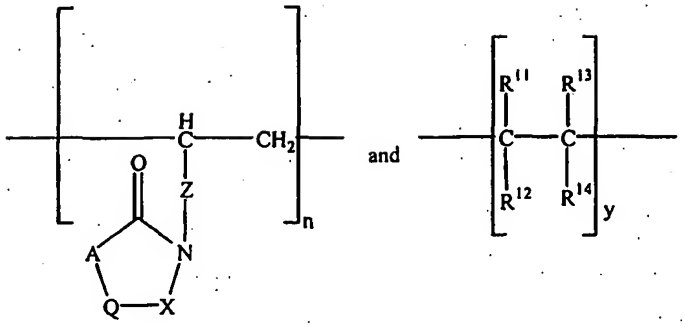
12. The multifunctional textile composition of claim 1, wherein said antimicrobial functionality is imparted with a member selected from the group consisting of a heterocyclic N-halamine and an antimicrobial polymer.

13. The multifunctional textile composition of claim 12, wherein said antimicrobial functionality is imparted with a heterocyclic N-halamine.

14. The multifunctional textile composition of claim 13, wherein said heterocyclic N-halamine is a halogenated product of a member selected from the group consisting of monomethylol-5,5-dimethylhydantoin (MDMH), 1,3-dimethylol-5,5-dimethylhydantoin (DMDMH); monomethylolated and dimethylolated derivatives of 2,2,5,5-tetramethyl-1,3-imidazolidin-4-one, 6,6-dimethyl-1, 3,5-triazine-2, 4-dione, 4,4,5,5-tetramethyl-1,3-imidazolidin-2-one, cyanuric acid and 5,5-dimethylhydantoin; and monomethoxylated and dimethoxylated derivatives of monomethylolated and dimethylolated derivatives of 2,2,5,5-tetramethyl-1,3-imidazolidin-4-one, 6,6-dimethyl-1,3,5-triazine-2,4-dione, 4,4,5,5-tetramethyl-1,3-imidazolidin-2-one, cyanuric acid, 5,5-dimethylhydantoin.

15. The multifunctional textile composition of claim 12, wherein said antimicrobial functionality is imparted with an antimicrobial polymer.

16. The multifunctional textile composition of claim 15, wherein said antimicrobial polymer comprises a mixture of monomeric units having the formulae:



I

wherein:

A is a member selected from the group consisting of NH, N-R^8 and CR^1R^2, wherein R^8 is a halogen;

R^1 and R^2, are each independently selected from the group consisting of optionally substituted (C_1-C_6)alkyl, optionally substituted (C_2-C_6)alkenyl, optionally substituted (C_2-C_6)alkynyl, optionally substituted cycloalkyl, optionally substituted (C_1-C_6)alkoxy, optionally substituted aryl and optionally substituted heteroaryl;

or, R^1 and R^2 and the carbon to which they are bound join to form an optionally substituted carbocyclic or optionally substituted heterocyclic ring;

Q is a member selected from the group consisting of C(O), NH, N-R^9 and CR^3R^4, wherein R^9 is a halogen;

R^3 and R^4, are each independently selected from the group consisting of optionally substituted (C_1-C_6)alkyl, optionally substituted (C_2-C_6)alkenyl, optionally substituted (C_2-C_6)alkynyl, optionally substituted cycloalkyl, optionally substituted (C_1-C_6)alkoxy, optionally substituted aryl and optionally substituted heteroaryl;

or, R^3 and R^4 and the carbon to which they are bound, join to form an optionally substituted carbocyclic or optionally substituted heterocyclic ring.

X is a member selected from the group consisting of C(O)-NR^{10} and CR^6R^7, wherein R^{10} is a member selected from the group consisting of hydrogen, halogen, optionally substituted (C_2-C_6)alkenyl and optionally substituted (C_1-C_6)alkyl;

R^6 and R^7, are each independently selected from the group consisting of optionally substituted (C_1-C_6)alkyl, optionally substituted (C_2-C_6)alkenyl, optionally substituted (C_2-C_6)alkynyl, optionally substituted cycloalkyl, optionally substituted (C_1-C_6)alkoxy, optionally substituted aryl and optionally substituted heteroaryl;

or, R^6 and R^7 and the carbon to which they are bound join to form an optionally substituted carbocyclic or optionally substituted heterocyclic ring;

Z is a member selected from the group consisting of optionally substituted (C_1-C_3)alkylene, C(O), or a single bond;

R^{11} is a member selected from the group consisting of hydrogen, halogen, hydroxyl, cyano, (C_1-C_6)alkyl, (C_2-C_6)alkenyl, (C_1-C_6)alkoxy, (C_1-C_6)alkylcarbonyl, (C_1-C_6)alkylcarboxyl, aldehydo, amido, aryl and heterocyclyl;

R^{12} is a member selected from the group consisting of hydrogen, halogen, hydroxyl, cyano, (C_1-C_6)alkyl, (C_2-C_6)alkenyl, (C_1-C_6)alkoxy, (C_1-C_6)alkylcarbonyl, (C_1-C_6)alkylcarboxyl, aldehydo, amido, aryl and heterocyclyl;

R^{13} is a member selected from the group consisting of hydrogen, halogen, hydroxyl, cyano, (C_1-C_6)alkyl, (C_2-C_6)alkenyl, (C_1-C_6)alkoxy, (C_1-C_6)alkylcarbonyl, (C_1-C_6)alkylcarboxyl, aldehydo, amido, aryl and heterocyclyl;

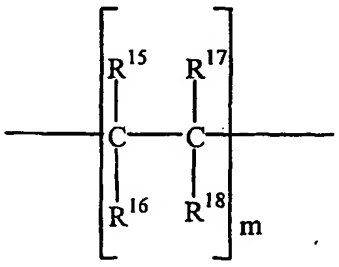
R^{14} is a member selected from the group consisting of hydrogen, halogen, hydroxyl, cyano, (C_1-C_6)alkyl, (C_2-C_6)alkenyl, (C_1-C_6)alkoxy, (C_1-C_6)alkylcarbonyl, (C_1-C_6)alkylcarboxyl, aldehydo, amido, aryl and heterocyclyl; and

n and y are each independently an integer from 1 to 250 inclusive.

17. The multifunctional textile composition of claim 16, wherein: n is 1 and y is 1.

18. The multifunctional textile composition of claim 16, wherein: A is NH and Z is CH_2.

19. The multifunctional textile composition of claim 15, wherein said antimicrobial polymer comprises at least one functional monomeric unit having the formula:



III

wherein:

R^{15}, R^{16} and R^{17} are each independently, a member selected from the group consisting of hydrogen, halogen, hydroxyl, cyano, (C_1-C_6)alkyl, (C_2-C_6)alkenyl, (C_1-

C_6)alkoxy, (C_1-C_6)alkylcarbonyl, (C_1-C_6)alkylcarboxyl, aldehydo, amido, aryl and heterocyclyl;

R^{18} is a member selected from the group consisting of $-CO_2^- X^{1+}$, $SO_3^- X^{1+}$, $-O^- X^{1+}$, $-PO_4^{-2} Z X^{1+}$ and $-PO_3^{-2} Z X^{1+}$;

X^1 is a member selected from the group consisting of a quaternary ammonium salt, a basic dye, and a mixture thereof;

Z^1 is a member selected from the group consisting of hydrogen and an alkaline earth metal; and

m is an integer from 1 to 250 inclusive.

20. The antimicrobial polymer according to claim 19, wherein said polymer is a long-chain synthetic acrylic polymer or fiber comprising at least 35% by weight of acrylonitrile units.

21. The antimicrobial polymer according to claim 19, wherein said polymer is a nylon.

22. A process for preparing a multifunctional textile, said process comprising:

(a) preparing a textile having an antimicrobial functionality to generate an antimicrobial textile; and

(b) treating said antimicrobial textile with a chemical agent to impart an additional functionality, thereby preparing said multifunctional textile.

23. The process of claim 22, wherein said additional functionality is a member selected from the group consisting of waterproofing, soil repellent finishing, fire resistance finishing, wrinkle free finishing, anti-UV finishing, and antistatic and softening finishing.

24. The method of claim 23, wherein said additional functionality is a waterproofing finishing.

25. The method of claim 24, wherein said waterproofing finishing is imparted with a fluorocarbon agent or silicone agent.

26. The method of claim 23, wherein said additional functionality is a fire resistant finishing.

27. The method of claim 26, wherein said fire resistance finishing is imparted with a agent selected from the group consisting of a phosphorus agent, a nitrogen agent, a bromine agent, an antimony agent and combinations thereof.

28. The method of claim 23, wherein said additional functionality is a wrinkle free finishing.

29. The method of claim 23, wherein said additional functionality is an anti-UV finishing.

30. The method of claim 29, wherein said anti-UV finishing is imparted with agent selected from the group consisting of an ultraviolet absorbent or a light stabilizer.

31. The method of claim 23, wherein said additional functionality is an antistatic and softening finishing.

32. The method of claim 22, wherein said textile is a member selected from the group consisting of cellulosic, cellulosic-polyester, polyester, nylon, polypropylene, acrylics, cotton, wool, silk, polyamide, aramid, olefin, spandex, vinyon, vinyl, graphite, and combinations and blends thereof.

33. The method of claim 22, wherein said antimicrobial functionality is imparted with a member selected from the group consisting of a heterocyclic N-halamine and an antimicrobial polymer.

34. The method of claim 33, wherein said antimicrobial functionality is imparted with a heterocyclic N-halamine.

35. The method of claim 33, wherein said antimicrobial functionality is imparted with an antimicrobial polymer.

36. The method of claim 34, wherein said heterocyclic N-halamine is a halogenated product of a member selected from the group consisting of monomethylol-5,5-dimethylhydantoin (MDMH), 1,3-dimethylol-5,5-dimethylhydantoin (DMDMH);

monomethylolated and dimethylolated derivatives of 2,2,5,5-tetramethyl-1,3-imidazolidin-4-one, 6,6-dimethyl-1,3,5-triazine-2,4-dione, 4,4,5,5-tetramethyl-1,3-imidazolidin-2-one, cyanuric acid and 5,5-dimethylhydantoin; and monomethoxylated and dimethoxylated derivatives of monomethylolated and dimethylolated derivatives of 2,2,5,5-tetramethyl-1,3-imidazolidin-4-one, 6,6-dimethyl-1,3,5-triazine-2,4-dione, 4,4,5,5-tetramethyl-1,3-imidazolidin-2-one, cyanuric acid, and 5,5-dimethylhydantoin.

37. A garment, said garment comprising:

an antimicrobial functionality; and

a chemical agent attached thereto to impart an additional functionality.

38. The garment of claim 37, wherein said additional functionality is a member selected from the group consisting of a waterproof finishing, soil repellent finishing, fire resistance finishing, wrinkle free finishing, anti-UV finishing, and antistatic finishing.

39. The garment of claim 37, wherein said garment is selected form the group consisting of a surgeon's gown, a cap, a mask, a surgical cover, a patient drape, carpeting, a bedding material, underwear, a sock and a uniform.

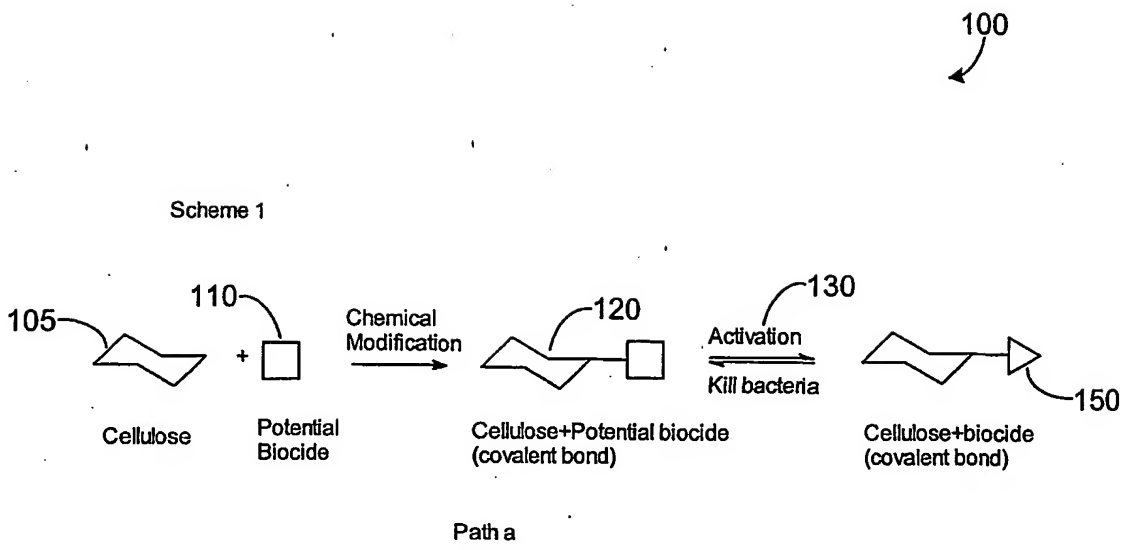
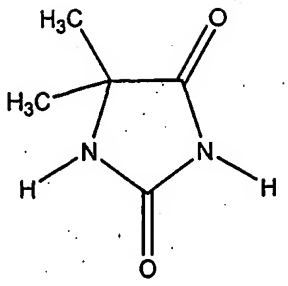
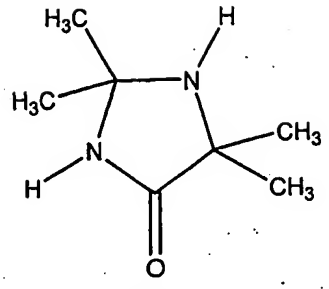

100
Scheme 1
105
110
+
Chemical
Modification
120
130
Activation
Kill bacteria
150
Cellulose
Potential
Biocide
Cellulose+Potential biocide
(covalent bond)
Cellulose+biocide
(covalent bond)
Path a

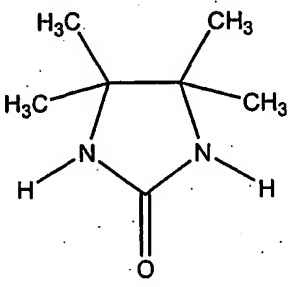
FIG. 1



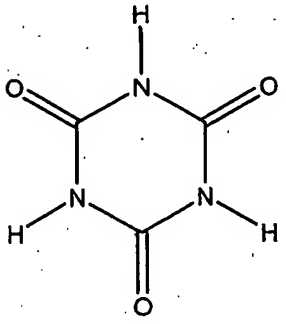
5,5-Dimethylhydantoin



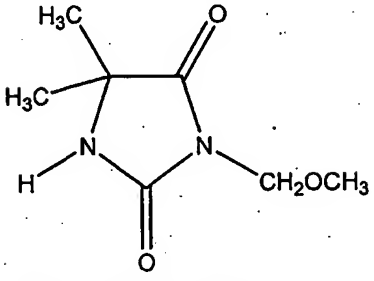
2,2,5,5-Tetramethyl-
4-imidazolidinone



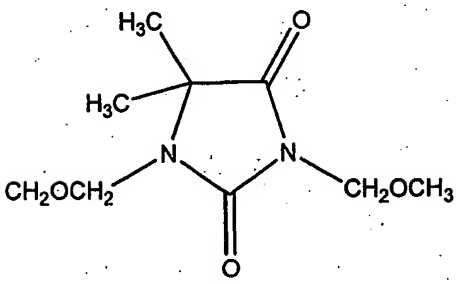
4,4,5,5-Tetramethyl-
2-imidazolidinone



Triazine-1,3,5-trione



Monomethoxymethyl-
5,5-dimethylhydantoin



1,3-Dimethoxymethyl-
5,5-dimethylhydantoin

FIG. 2

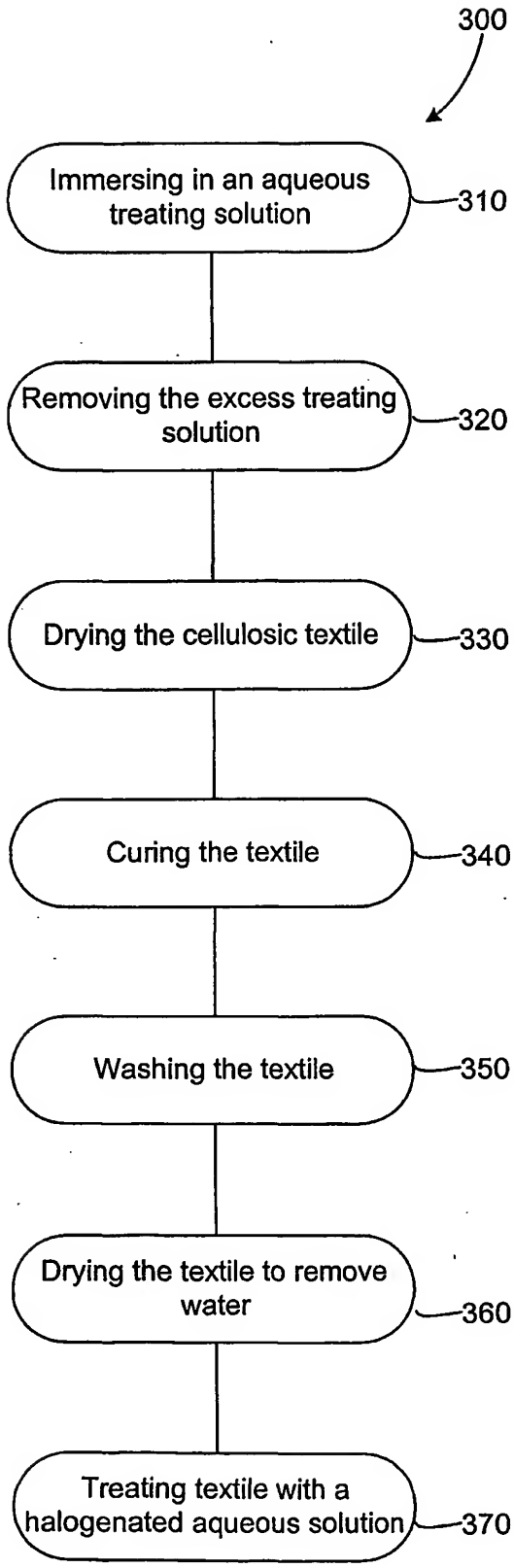

300
Immersing in an aqueous treating solution
310
Removing the excess treating solution
320
Drying the cellulosic textile
330
Curing the textile
340
Washing the textile
350
Drying the textile to remove water
360
Treating textile with a halogenated aqueous solution
370

FIG. 3

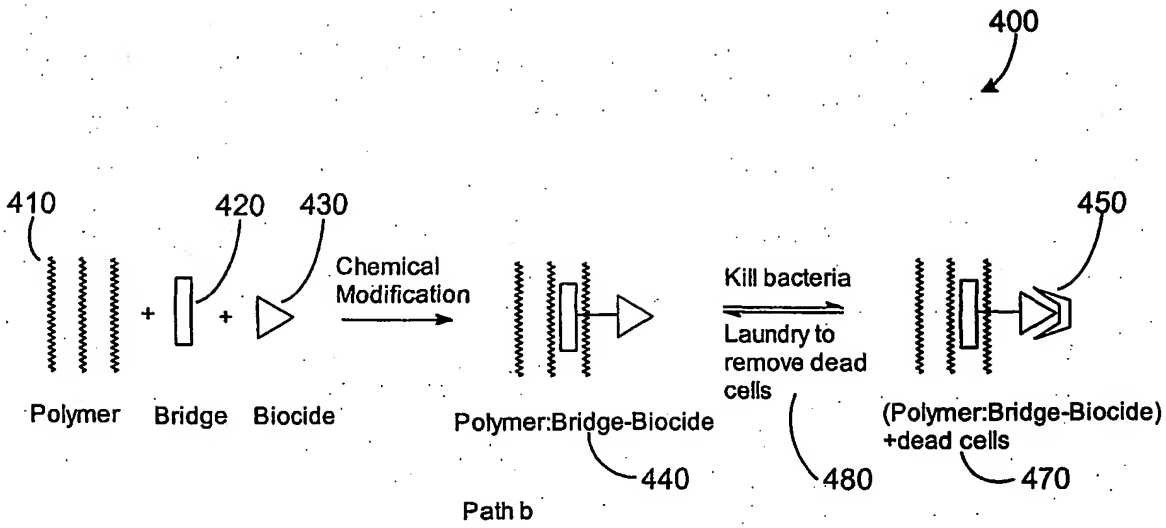
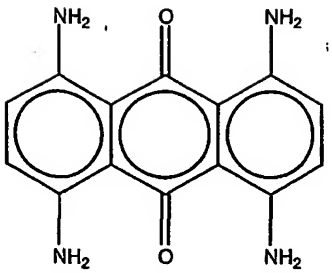
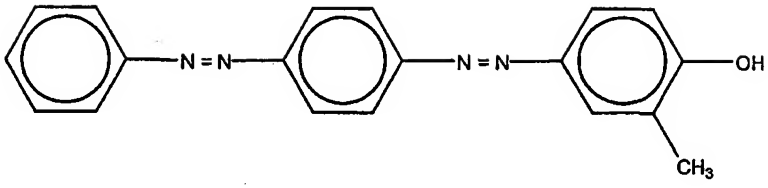

400
410
420
430
450
Chemical Modification
Kill bacteria
Laundry to remove dead cells
Polymer
Bridge
Biocide
Polymer:Bridge-Biocide
(Polymer:Bridge-Biocide) +dead cells
440
480
470
Path b

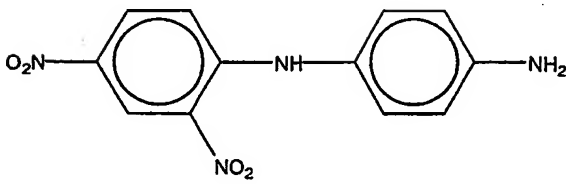
FIG. 4



DISPERSE BLUE 1



DISPERSE YELLOW 7



DISPERSE YELLOW 9

FIG. 5

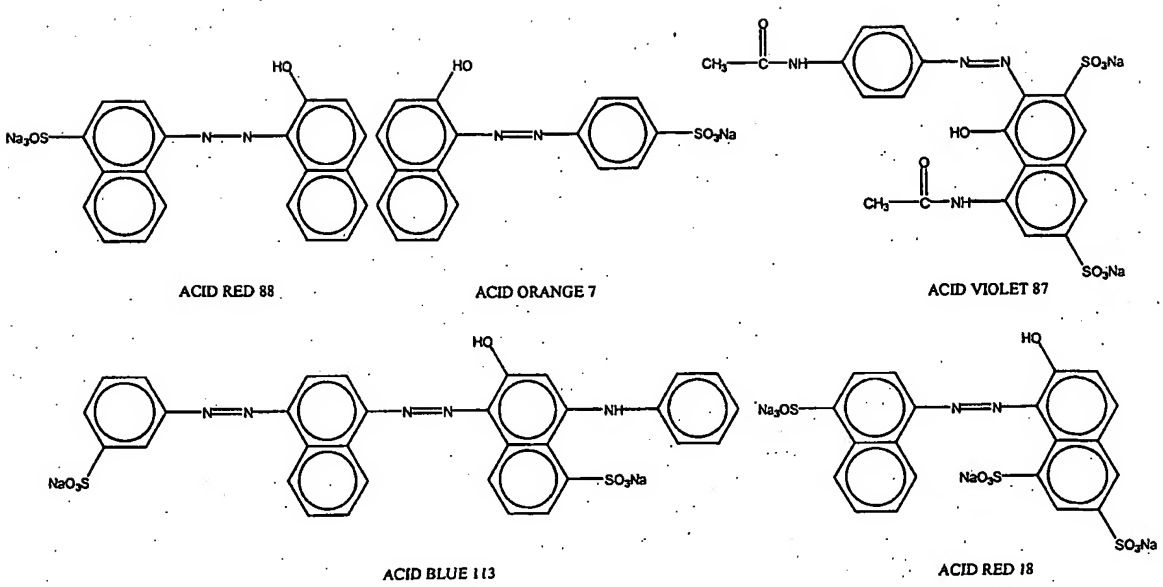

HO
Na3OS
N=N
SO3Na
CH3
C
NH
O
ACID RED 88
ACID ORANGE 7
ACID VIOLET 87
NaO3S
ACID BLUE 113
ACID RED 18

FIG. 6

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US02/09788

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : D06M 13/352, 13/355, 13/364.
US CL : 8/189, 8/181, 8/190, 8/191, 8/115.7, 252/8.84, 252/8..86,424/405;

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
U.S. : 8/189, 8/181, 8/190, 8/191, 8/115.7, 252/8.84, 252/8..86,424/405;

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5,670,646 A (WORLEY et al) September 23, 1997, column 5, line46 - column 16, line 53 and col. 30, lines 24-63.	1-39
X	US 5,882,357 A (SUN et al) 16 March 1999, column 3, line56 - column 7, line 34.	1-39
X,P	US 6,077,319 A (SUN et al) 20 June 2000., entire document.	1-39

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search
28 June 2002 (28.06.2002)

Date of mailing of the international search report
31 JUL 2002

Name and mailing address of the ISA/US
Commissioner of Patents and Trademarks
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Telephone No. 703 308-0661

Form PCT/ISA/210 (second sheet) (July 1998)